REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. REPORT DATE *(DD-MM-YYYY)*	**2. REPORT TYPE** Technical Papers	**3. DATES COVERED** *(From - To)*
4. TITLE AND SUBTITLE		**5a. CONTRACT NUMBER**
		5b. GRANT NUMBER
		5c. PROGRAM ELEMENT NUMBER
6. AUTHOR(S)		**5d. PROJECT NUMBER**
		5e. TASK NUMBER
		5f. WORK UNIT NUMBER
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Air Force Research Laboratory (AFMC) AFRL/PRS 5 Pollux Drive Edwards AFB CA 93524-7048		**8. PERFORMING ORGANIZATION REPORT**
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) Air Force Research Laboratory (AFMC) AFRL/PRS 5 Pollux Drive Edwards AFB CA 93524-7048		**10. SPONSOR/MONITOR'S ACRONYM(S)**
		11. SPONSOR/MONITOR'S NUMBER(S)

12. DISTRIBUTION / AVAILABILITY STATEMENT

Approved for public release; distribution unlimited.

13. SUPPLEMENTARY NOTES

14. ABSTRACT

20020830 099

15. SUBJECT TERMS

16. SECURITY CLASSIFICATION OF:			**17. LIMITATION OF ABSTRACT**	**18. NUMBER OF PAGES**	**19a. NAME OF RESPONSIBLE PERSON** Leilani Richardson
a. REPORT Unclassified	**b. ABSTRACT** Unclassified	**c. THIS PAGE** Unclassified	A		**19b. TELEPHONE NUMBER** *(include area code)* (661) 275-5015

Standard Form 298 (Rev. 8-98)
Prescribed by ANSI Std. 239.18

5 items enclosed

FILE

MEMORANDUM FOR PRS (In-House Publication)

FROM: PROI (STINFO) 19 July 2002

SUBJECT: Authorization for Release of Technical Information, Control Number: **AFRL-PR-ED-TP-2002-187**
Wesley Hoffman (PRSM), "In Situ Rapid Densification Utilizing a Low-Viscosity Wetting Impregnant That Greatly Reduces Processing Time to Produce Uniform Density Carbon-Carbon Composites"

55768

AFRL Horizons Magazine **(Statement A)**
(Deadline: None given)

In Situ Rapid Densification Utilizing a Low-Viscosity Wetting Impregnant That Greatly Reduces Processing Time to Produce Uniform-Density Carbon–Carbon Composites

High-performance carbon–carbon (C–C) composites possess a unique set of properties that make them desirable materials for high-temperature structural uses such as in rocket propulsion components, hypersonic vehicles, and aircraft brakes. They are stronger and stiffer than steel and less dense than aluminum. In a non-oxidizing environment they maintain their mechanical properties to temperatures above 3000°C. In addition, their properties actually improve with heating as the non-ordered carbon is converted to the ordered graphite structure (the process of graphitization). Despite these properties, however, the use of carbon–carbon composites has been limited both because of their high cost and their oxidation at elevated temperatures. A recent development in composite processing at the Propulsion Directorate of the Air Force Research Laboratory (AFRL), Edwards, California—called *in situ* rapid densification—greatly reduces their processing time and their cost.

For ~30 years, carbon–carbon composites have been manufactured principally by two processes that use different means to place the matrix among the fibers in the preform and thus densify the composite. One process involves infiltrating the composite preform with a hydrocarbon gas which pyrolyzes to produce carbon. The other process involves impregnating the fiber preform with a liquid hydrocarbon such as petroleum or coal tar pitch or a resin followed by carbonization, which removes all non-carbon atoms. Both processes are followed by graphitization in which the partially densified composite is heated to temperatures above 2400°C to open up closed porosity in addition to enhancing mechanical properties.

After the composite is graphitized, all the present commercial processes require machining the outside of the partially densified composite to remove material. This step is required because all these processes preferentially densify the outside of the composite, which blocks the surface pores so that additional densification cannot occur. Because of this shortcoming, the infiltration/impregnation-carbonization-graphitization-machining cycle must be repeated at least three to ten times depending on the final desired density. Since each cycle requires 3–4 weeks, densification is time-consuming and costly. By understanding the reasons for the shortcomings of current commercial processing, the *in situ* rapid densification process developed at the Propulsion Directorate is able to avoid inhomogeneous densification and reduce the time required from many months to less than two weeks, thereby markedly decreasing the energy and labor cost.

This novel process rapidly densifies the composite uniformly because it addresses the two

opposing requirements of the carbon matrix precursor. On the one hand, an impregnant with low viscosity and good wettability is required to get the matrix precursor uniformly into the fiber perform. On the other hand a high-char-yield carbon precursor is required to minimize the number of densification cycles required. This improvement is accomplished by using a low-viscosity impregnant , such as naphthalene, that wets the fiber preform surface and is then absorbed into the fiber preform, like water into a sponge, completely and uniformly filling the preform. Thus, this process gives a uniform density and eliminates the need for machining after densification. After the impregnant has filled the preform, the impregnant is polymerized into a high-char-yield carbon-matrix precursor, which is then pyrolyzed to produce a uniform, dense high quality carbon matrix. Since the carbon-matrix precursor has a high carbon yield fewer cycles are needed to bring the composite to final density.

Such a uniformly densified composite can be produced from any type of fiber preform (e.g., woven, braided, 1-dimensional to *n*-dimensional, or felt). Furthermore, in contrast to other processes, there does not appear to be a size limitation; preforms with diameters up to 45 cm and lengths up to 183 cm have been uniformly densified. An additional advantage of this technology is the ability to join carbon–carbon parts with a mechanically strong, seamless joint. Some of the products produced by this process can be seen in Figure 1.

In this process, it is not necessary to graphitize the composites, thus saving time and energy. Even without graphitization, the ablation and erosion performance of the C–C composite produced by this process equals that of commercially produced composites of equal density that are exposed to the exhaust of liquid rocket engines or of solid rocket motors. In most applications, it is not necessary to graphitize the material densified by the In Situ process. However, when it is exposed to temperatures only slightly above 2000°C, a highly graphitic material with high thermal conductivity is produced. The significance of this result is that it is possible to use the low-cost liquid phase process to produce C–C composites with the properties of composites produced by a higher-cost gas-phase process. It is anticipated that the expected major decrease in processing costs associated with this process will result in many new applications for this material.

\Figure Caption

Figure 1. Components fabricated by the *in situ* rapid densification process include brakes, nozzles, pistons, hypersonic leading edges, heating elements, and structural components.

In Situ Rapid Densification with Low-Viscosity Impregnant Reduces Processing Time to Produce Uniform Density Carbon–Carbon Composites

High-performance carbon–carbon (C–C) composites possess a set of properties that make them desirable materials for high-temperature structural uses such as in rocket propulsion components, hypersonic vehicles, and aircraft brakes. They are stronger and stiffer than steel and less dense than aluminum. In addition, they maintain their mechanical properties to temperatures above 3000°C, and their properties improve with heating as the nonordered carbon is converted to the ordered graphite structure (the process of graphitization). Despite these properties, however, the use of C–C composites has been limited both because of their high cost and their oxidation at elevated temperatures. A recent development in composite processing at the Air Force Research Laboratory (AFRL), Edwards, California—called *in situ* rapid densification—greatly reduces their processing time and thus their cost.

For ~30 years, C–C composites have been manufactured principally by two processes that use different means to combine the matrix and fibers and to densify the composite. One process involves impregnating the fiber preform with a liquid hydrocarbon such as pitch or a resin followed by carbonization, which removes all noncarbon atoms. The other process involves infiltrating the composite preform with a hydrocarbon gas. Both processes are followed by graphitization in which the partially densified composite is heated to temperatures above 2400°C to enhance its mechanical properties.

After the composite is graphitized, these processes require machining the outside of the partially densified composite to remove material. This step is required because both processes preferentially densify the outside of the composite, which blocks the surface pores so that additional densification cannot occur. Because of this shortcoming, the infiltration/impregnation-carbonization-graphitization-machining cycle must be repeated at least three times. Since each cycle requires 3–4 weeks, densification is time-consuming and costly. By understanding the reasons for the shortcomings of current commercial processing, the *in situ* rapid densification process is able to avoid inhomogeneous densification and reduce the time required from many months to less than two weeks, thereby markedly decreasing the production cost.

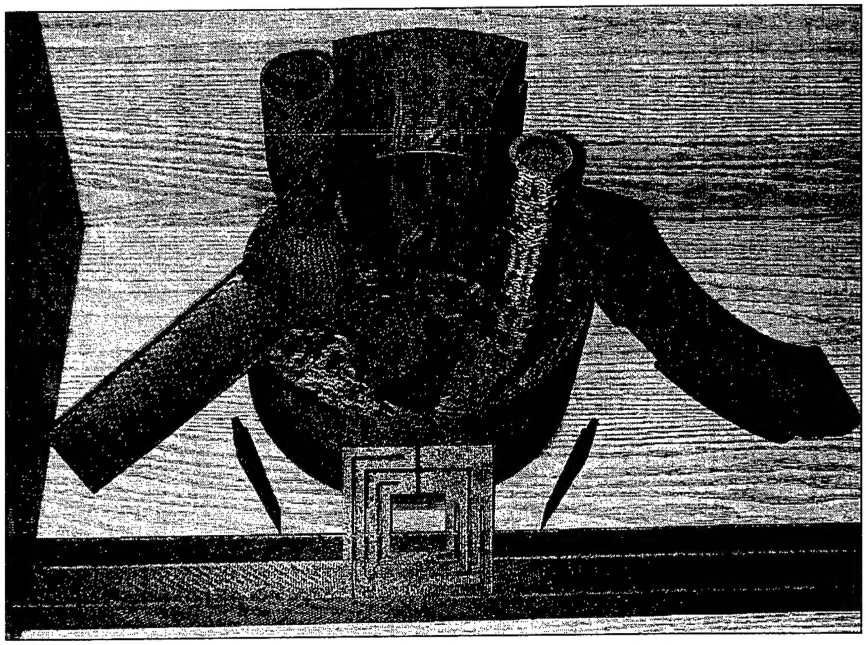

Figure 1. Components fabricated by the in situ *rapid densification process include brakes, nozzles, pistons, hypersonic leading edges, heating elements, and structural components.*

This novel process rapidly densifies the composite uniformly because it addresses the opposing requirements of the impregnant's low viscosity and good wettability on the one hand and the need for a high-char-yield carbon precursor on the other. This improvement is accomplished by using a low-viscosity impregnant, such as naphthalene, that wets the fiber preform surface and is then absorbed into the fiber perform, like water into a sponge, completely and uniformly filling the preform. Thus, this process gives a uniform density and eliminates the need for machining after densification. After the impregnant has filled the preform, the impregnant is polymerized into a carbon-matrix precursor, which is then pyrolyzed to produce a uniform, dense carbon matrix. Since the carbon-matrix precursor has a high carbon yield, fewer cycles are needed to bring the composite to final density.

Such a uniformly densified composite can be produced from any type of fiber preform (e.g., woven, braided, one-dimensional to n-dimensional, felt). Furthermore, in contrast to other processes, there does not appear to be a size limitation; preforms with diameters up to 45 cm and lengths up to 183 cm have been uniformly densified. An additional advantage of this technology is the ability to join C–C parts with a mechanically strong, seamless joint. Some of the products produced by this process can be seen in Figure 1.

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Opportunities

The AFRL High Temperature Components Group welcomes inquiries in cooperative research and development.

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